June 2003 Editor: Regina R. Montgomery

SRM SPOTLIGHT

In this Issue…

New SRMs

SRM 2133 Phosphorus Implant in Silicon Depth Profile Standard

The U.S. semiconductor industry relies heavily on secondary ion mass spectrometry (SIMS) for characterization of the depth distribution of dopants in silicon. Increasingly stringent requirements are being placed on these measurements according to the International Technology Roadmap for Semiconductors (ITRS).

To achieve high accuracy in the concentration determination by SIMS, standards of known dopant concentration are required. These requirements are conveniently satisfied by ion implants of certified dose.

Standard Reference Materials of boron (SRM 2137) and arsenic (SRM 2134) implants in silicon were previously issued by NIST for calibration of these common dopant elements. In a survey of their Analytical Laboratory Managers' Council, SEMATECH also listed a reference material for phosphorus in silicon as a high priority requirement, in addition to boron and arsenic. NIST responded to this need and has now issued SRM 2133 – Phosphorus Implant in Silicon Depth Profile Standard.

The certification measurements were made by radiochemical neutron activation analysis. Measurements on individual pieces of silicon containing less than 50 ng of phosphorus resulted in a certified value of 9.58×10^{14} atoms/cm^2 and an expanded relative uncertainty (approximate 95 % confidence interval) of ± 1.7 % that will satisfy the ITRS requirement. One unit of SRM 2133 consists of a 1 cm x 1 cm piece of an implanted silicon wafer.

Technical Contact: David S. Simons
Email: david.simons@nist.gov

New SRMs continued…

SRM 1917 Mercury Porosimetry Standard

After two years of extensive work, the "Agreement on Cooperation in the Field of Reference Materials" between NIST and Bundesanstalt fur Materialforschung und-prufung, (BAM) was successfully finished in August 2002. This is the first joint certification project between NIST and BAM. The material officially named NIST SRM 1917 / CRM BAM-P127 (pellets of Al_2O_3 with a diameter of about 1 mm) is a certified nanoporous reference material for mercury-porosimetry ("Mercury Porosimetry Standard").

Seventeen laboratories from the United States and 15 laboratories from the European Union took part in the interlaboratory comparison. The statistical evaluation (calculation of the mean intrusion curve with uncertainty boundaries) was carried out at NIST using the Vangel Rukhin algorithm.

Certified Properties

a) the mercury intrusion curve between 0.1 MPa and 400 MPa
b) the cumulative pore volume curve between 3.7 nm and 14708 nm
c) the pore volume at selected intrusion pressures as well as the mean and the most frequent pore diameter.

This first joint development and certification of a reference material represents a new form of collaboration between NIST and BAM. This development avoids duplication of effort on an international scale and therefore saves time and money. Also, the international effort ensures a better acceptance for this reference material compared to materials that have merely been certified by the national authority of just one country.

SRM Contact: Bruce MacDonald
E-Mail: bruce.macdonald@nist.gov

New SRMs continued…

Hard Rock Mine Waste SRM 2780

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparati used to determine heavy metals and other elements in hard rock mine waste and materials of a similar matrix. SRM 2780 is composed of material collected from a waste pile of an abandoned mine site near Silverton, CO.

Certified mass fraction values for 12 elements are listed in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. All values are reported as mass fractions [1], on a dry mass basis (see *Instructions for Drying* as shown on the certificate), and are based on measurements using a sample mass of at least 250 mg.

Table 1. Certified Mass Fractions

Element	Mass Fraction (%)	*k*
Aluminum	8.87 ± 0.33	2.3
Calcium	0.195 ± 0.020	2.6
Iron	2.784 ± 0.080	2.4
Lead	0.577 ± 0.041	2.4
Magnesium	0.533 ± 0.020	2.8
Potassium	3.38 ± 0.26	2.8
Sodium	0.221 ± 0.018	2.8
Sulfur	1.263 ± 0.042	2.6
Zinc	0.257 ± 0.016	2.6

Element	Mass Fraction (mg/kg)	*k*
Arsenic	48.8 ± 3.3	2.0
Cadmium	12.10 ± 0.24	2.8
Mercury	0.710 ± 0.042	2.6

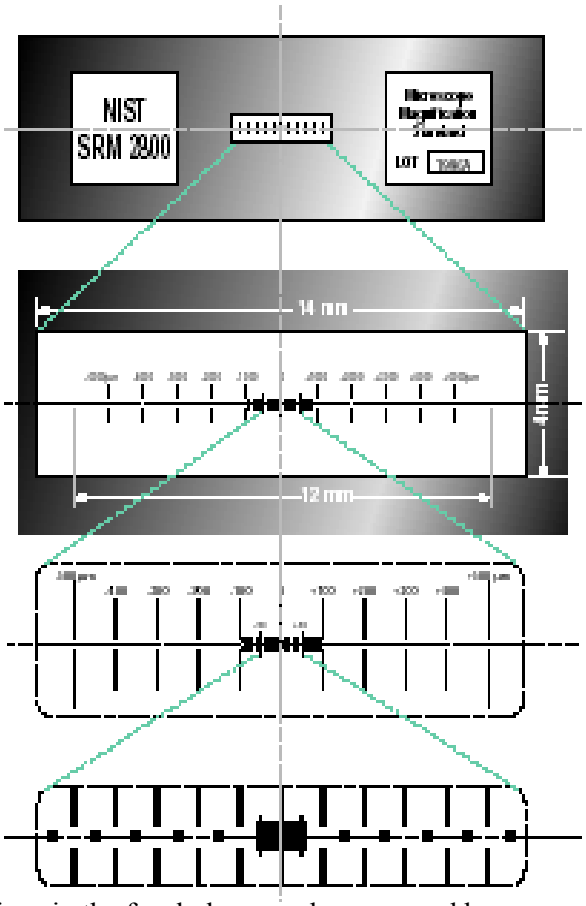
Technical Contact: Gregory C. Turk
E-Mail: gregory.turk@nist.gov

New SRMs continued…

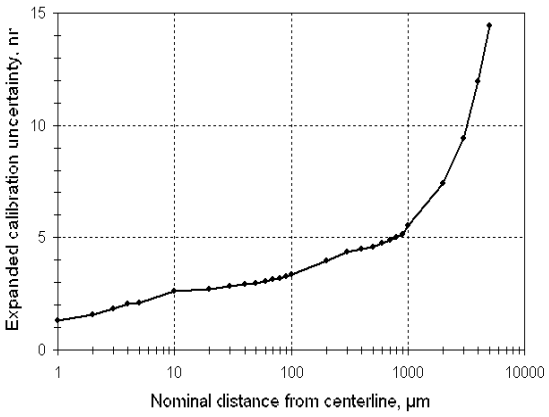
SRM 2800 Microscope Magnification Standard

Exciting new advances are being made every day in the field of nanotechnology. These developments promise to improve the quality of life for all of us. In the course of this research and development, it frequently becomes necessary to measure the size of a "nano-object," and this can be done only with the aid of a microscope. Optical microscopes in reflection or transmission, scanning electron (SEMs), and atomic force (AFM) microscopes and other scanning probe-type microscopes are popular tools for this purpose.

The magnification of any type of microscope can be calibrated for these measurements using SRM 2800. This SRM consists of a pattern of parallel lines whose nominal distances from the centerline range from 1 μm to 5 mm (see figure at right). The pattern is printed in etched chrome on a fused quartz substrate with nominal dimensions of 25 × 75 × 2.3 mm (1 × 3 × 0.09 in.) using photomask production techniques. The pattern is designed so the major lines are visible at low magnification and wide field of view, and the smaller lines become more visible at progressively higher magnifications, without having to recenter the sample. Certified values are given for the center-to-center distance of each line from the centerline; the linewidths are not certified.



The scale or magnification of a microscope image along various directions in the focal plane can be measured by rotating the SRM, and scale linearity throughout the field of view can be determined by repositioning the SRM in the object plane.



It is essential to reduce the effect of charging of the quartz substrate. Also, the scale of the microscope image of a nano-object can be accurately calibrated with SRM 2800, the positions of the object's edges in the image can be difficult to identify. Note that this SRM is slightly thicker than a standard microscope slide.

Every line on every SRM unit is individually calibrated. The expanded calibration uncertainty ranges from less than 5 nm for line spacing below 1 mm to ~15 nm at the 5 mm spacing. The aggregate expanded (2σ) calibration uncertainty for all of the SRM 2800s produced to date is shown in the figure at left. The materials used for this SRM-fused quartz and oxidized chrome are dimensionally stable and relatively chemically inert. Consequently, the certification is deemed to be valid indefinitely if the SRM is kept clean and used with care.

Technical Contact: James E. Potzick
E-Mail: james.potzick@nist.gov

New SRMs continued…

Standard Reference Material 1727 Anode Tin

Anode tin is used as a raw material in tin-plating lines that make tinplate (tinplated sheet steel) for the container industry. Tinplate is a high volume commodity made by many US and foreign steel companies. About 3 million tons of tinplate are manufactured and shipped from US steel producers annually, at a typical selling value of $800 per ton during 1999. Can makers purchase much of the tinplate to manufacture products such as food and beverage cans, automotive filter cases, and paint cans.

Steel mills typically purchase tin anodes according to ASTM B 339 Standard Specification for Pig Tin, with a restriction of 50 ppm lead maximum. In recent years, can makers reduced the allowable lead content of tin plate in recognition of potential health risks in products used for foods. The anodes are electrochemically dissolved, and the tin is plated onto sheet steel. Residual amounts of anode tin are typically remelted on-site to form new anodes. Any lead in the plating bath co-deposits with the tin on the steel. Incoming and remelted tin anodes are analyzed by atomic emission spectrometry to meet material acceptance and process control requirements.

Standard Reference Material 1727 Anode Tin is intended primarily for use in evaluating chemical and instrumental methods of analysis of refined pig tin for lead content. SRM 1727 is sold in solid form for spectrometric analysis and may be chipped for use with chemical methods of analysis. SRM 1727 is certified for lead content on the basis of analyses by isotope-dilution inductively-coupled plasma mass spectrometry. Information values are provided for ten additional elements.

Table 1. Certified Values for SRM 1727 Anode Tin

Constituent	Mass Fraction (mg/kg)
Pb (lead)	33.26 ± 0.33

A unit of SRM 1727 consists of a block of Grade “A” tin for the manufacture of tinplate with dimensions approximately 30 mm × 30 mm × 30 mm.

Technical Contact: John Sieber
E-Mail: john.sieber@nist.gov

New SRMs continued…

SRM 1932 Fluorescein Solution

Fluorescence-based assays have become very popular because of their high sensitivity and selectivity. For a variety of reasons, measurements of fluorescence intensity are much more difficult to quantitate than determinations of other optical properties such as absorbance. Flow cytometry is a rapidly growing technique used to measure the fluorescence intensity of labeled particles or cells. By utilizing a coaxial flow technique, suspended particles are paraded, single file, past a laser beam that excites their fluorophore labels. The resulting fluorescence intensity is measured by one or more single wavelength filter fluorometers. In some cases, downstream flow diversion techniques are used to sort the particles according to their fluorescence signals (free chromosomes can be separated this way). It is particularly important to be able to determine the fluorescence intensity of the particles as they pass through the flow cytometer, as this may determine the number of bound fluorophores on the particle. This in turn may be a measure of the number of biologically active sites and may be of clinical importance, e.g., determining the number of antibodies bound to a cell.

Fluorescently labeled microspheres have long been used in flow cytometry as surrogates for cells and other biological particles. Such microbeads could be used to calibrate the fluorescence intensity measurements, if the precise number of fluorophores per microbead were known. Unfortunately, this is not an easy determination. Complicating this measurement problem is the sensitivity of fluorescence intensity to the immediate environment of the fluorophore. Factors such as pH, temperature, ionic strength, and the presence, absence, and concentration of other species in solution can alter fluorescence emission intensity, often significantly.

A concept known as "molecules of equivalent soluble fluorophores" or MESF provides a two step process that can circumvent these measurement problems and provide a quantitative calibration for flow cytometers. SRM 1932 *Fluorescein Solution* was certified for this purpose. The MESF concept relies on the equivalency between the number of fluorophores in two samples—one a solution of the fluorophore (fluorescein in this case) and the other a suspension of fluorescein-labled microbeads. It is important to realize that the microbeads are so small that they remain readily suspended and behave in most respects as though they were in solution. It is also significant that while the fluorophores are not identical (free fluorescein in one case and a fluorescein substructure bound to an uncoated particle with a linking chain in the other), their spectral behavior is nearly identical. For a given set of conditions, by using solutions containing known amounts of SRM 1932, a fluorescence-emission-intensity-calibration curve can be determined using a conventional spectrofluorometer. Then using a suspension of labeled microbeads under as nearly identical conditions as possible, the average number of equivalent fluorophores on the beads (i.e., their MESF value) can be determined. The assignments of MESF values to a set of labeled microbeads with different fluorophore densities allows for the construction of a calibration curve for a flow cytometer. It is only necessary to know the concentration of the fluorescein solutions used to make up the original fluorescence-intensity-calibration curve and to maintain identical environmental conditions for the fluorescein solutions and microbead suspensions. The utility of the MESF concept is not restricted solely to microbeads, as MESF values can be determined for other labeled particles such as lymphocytes and other biological entities.

SRM 1932 is certified for the concentration of fluorescein of a certified purity in a borate buffer solution. This SRM is provided as a solution to eliminate difficulties and subsequent errors in weighing very small amounts of this highly fluorescent material. (Non-spectroscopists may recognize fluorescein as the bright yellow-green colorant in fluorescent "high-lighter" marking pens.)

The mass fraction (purity) of the fluorescein material used to prepare this SRM is 97.55 % ± 0.64 %. The certified concentration of the fluorescein in SRM 1932 is 60.97 μmol·kg-1 ± 0.40 μmol·kg-1.

New SRMs continued…

Each unit of SRM 1932 consists of three sealed amber glass ampoules containing a solution of fluorescein in an aqueous borate buffer. Approximately 2.0 mL of the solution is flame-sealed into each individual ampoule that has been pre-scored for easy opening. The ampouled SRM material is sufficiently concentrated that it is expected to be highly diluted with the same material used to suspend the micro-particles prior to use thereby creating the essentially identical environmental conditions necessary for the MESF concept to work.

Technical Contacts: Gary Kramer, Adolfas Gaigalas, Paul DeRose, Lili Wang
E-Mail: gary.kramer@nist.gov, adolfas.gaigalas@nist.gov, paul.derose@nist.gov; lili.wang@nist.gov

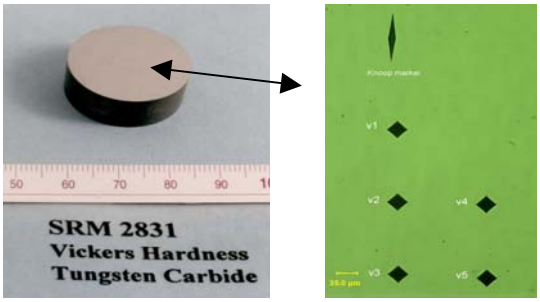
---

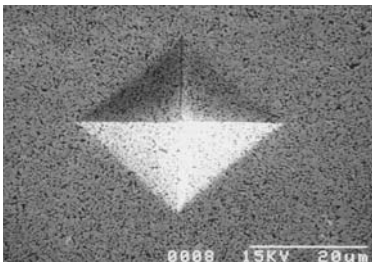
2831 Vickers Hardness of Ceramics and Hardmetals

The SRM is a hot-isostatically pressed tungsten carbide / 12% cobalt disk. The SRM has 5 indentations made at a load of 9.8 N (1 kgf) in the center of a polished face. The typical hardness HV1 is of the order of 15.0 Gpa or 1,530 kgf/mm^2. This is in middle of the hardness range for most ceramics and cutting tool carbides. The hardness is extremely consistent across the polished surface. Each SRM is individually certified for the size of each of the 5 indentations, the average diagonal length (typically 35.0 μm), and the average hardness measured from the 5 indentations. Each block is individually certified with the values on an accompanying certificate.

The SRM may be used with conventional hardness and microindentation hardness testing machines with optical microscopes. All indentation diagonal sizes were measured to within 0.1 μm with a calibrated optical research laboratory microscope with a high-resolution digital camera. Diagonal lengths in prototype disks were verified by calibrated scanning electron microscope measurements. An international round robin verified that consistent and accurate readings could be obtained. This SRM may be used in conjunction with:

ASTM C 1327	Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics
ASTM E 384	Microindentation Hardness of Materials
ASTM E 92	Vickers Hardness of Metallic Materials
ISO 14705	Fine Ceramics, Test Method for Hardness at Room Temperature
ISO 3878	Hardmetals – Vickers Indentation Test
CEN ENV 843-5	Advanced Technical Ceramics, Vickers, Knoop and Rockwell Superficial Hardness Tests
JIS R 1610	Testing Method for Vickers Hardness of High Performance Ceramics





SRM 2831 consists of a 25 mm D × 9.5 mm disk that has a nominal hardness of approximately 15.0 GPa (1530 kgf/mm^2) packaged in a wooden box. Each unit is individually certified and bears a serial number (the letter W followed by a hyphen and the disk number) scribed on the opposite (bottom) face of the disk.

Technical Contact: George Quinn
E-Mail: george.quinn@nist.gov

SRM News

SRM 2387 Peanut Butter
and
SRM 1946 Fish Tissue
are now available.

We now have our SRM Special Publications 260's posted on our website for your convenience.
http://www.nist.gov/srm

If you would like to receive your newsletter via e-mail, please send your e-mail address to:

Spotlight@nist.gov

NIST SRM Exhibit Schedule

Biotechnology International Convention & Exhibition (Bio 2003) - June 22-25, 2003, Washington, D.C.

American Association of Clinical Chemists (AACC) – July 20-24, 2003, Philadelphia, PA

American Chemical Society (ACS) - September 7-11, 2003, New York

Association of Analytical Communities (AOAC) – September 14-17, 2003, Atlanta Georgia

Eastern Analytical Symposium (EAS) – November 11-14, 2003, Somerset, New Jersey

Renewal SRMs

SRM 1845 Cholesterol in Whole Egg Powder

This Standard Reference Material (SRM) is intended primarily for use in evaluating the reliability of analytical methods used for the determination of cholesterol in whole egg material and similar food, and biological materials. SRM 1845 is one of a number of NIST reference materials available for evaluating the role of cholesterol in health and disease, establishing dietary requirements and recommendations for cholesterol, and accumulating accurate base-line and concentration data for cholesterol in foods.

The whole egg powder material used in SRM 1845 was obtained, prepared, and homogenized by Agriculture Canada. This material consists of Grade A (Canada) chicken eggs, dried with color and maximum 2 % Zeolex (sodium silicon aluminate) added as an anticaking agent. The material was radiation-sterilized in bulk and subsequently packaged in a Class 100 clean air hood at NIST.

The cholesterol concentration, expressed as a mass fraction in g/kg (mg/g) on an as received basis, was determined at NIST using a modification of the isotope dilution mass spectrometric (IDMS) definitive method for cholesterol.

Certified Cholesterol Concentration and Uncertainty

18.64 g/kg ± 0.39 g/kg

The certified concentration value and the uncertainty apply to a minimum sample size of 60 mg of the undried material. The uncertainty in the certified value, calculated according to the method described in the *ISO Guide* is expressed as an expanded uncertainty, U. The expanded uncertainty is calculated as $U = ku_c$, where uc is intended to represent, at the level of one standard deviation, the combined effect of within-method components of uncertainty and a component for observed material variability between bottles. The coverage factor, $k = 2$, corresponds to approximate 95 % confidence for each analyte.

SRM 1845 consists of one glass bottle, containing approximately 35 g of dried whole egg powder. This material is valid until 01 January 2007.

Technical Contact: Michael Welch
E-Mail: michael.welch@nist.gov

Other Renewals Now Available

SRM 723d	Tris Acidimetric
SRM 1021	Glass Beads-Particle Size Distribution
SRM 2034	Holmium Oxide Solution
SRM 2044a	White Diffuser
SRM 2491	Non-Newtonian Polymer Melt for Rhelogy
SRM 2627a	Nitric Oxide in Nitrogen 5 umol/mol
SRM 2628a	Nitric Oxide in Nitrogen 10 umol/mol

Renewals continued……

SRM 2636a	Carbon Monoxide in Nitrogen 250 umol/mol
SRM 2642a	Carbon Monoxide in Nitrogen 8% mol/mol
SRM 2750	Methane in Air 50 umol/mol
SRM 2751	Methane in Air 100 umol/mol
SRM 3103a	Arsenic Standard Solution
SRM 3184	Bromide Anion Standard Solution
SRM 3191	Aqueous Electrolytic Conductivity
SRM 3198	Aqueous Electrolytic Conductivity
SRM 3199	Aqueous Electrolytic Conductivity

Revisions - - - - - - - - Certificate Revisions – Are you Using These Materials?

Below is a list of our most recent certificate revisions. To gain maximum benefit from a NIST SRM, the certificate in possession must be current. NIST updates certificates for a variety of reasons, such as the extension of the certificate date or to include additional information gained from stability testing. If you do not have the most recent certificate for your material, download a copy from the website at: www.nist.gov/srm, or contact SRM at: telephone (301) 975-6776; fax (301) 926-4751 or email: srminfo@nist.gov.

SRM 1548a Typical Diet
New expiration date: This material is valid until 30 April 2004.

SRM 1563 Cholesterol and Fat-Soluble Vitamins in Coconut Oil
Removal of information values for retinyl acetate

SRM 1622e Sulfur in Residual Fuel Oil
New expiration date: This material is valid until 01 July 2015.

SRM 1658a Methane in Air 1 umol/mol
New expiration date: This material is valid until 01June 2008.

SRM 1963 Polystyrene Spheres
New expiration date: This material is valid until 31 January 2010.

SRM 2658 Oxygen in Nitrogen 10% mol/mol
New expiration date: This material is valid until 01 April 2008.

SRM 2647a Propane in Nitrogen 2500 umol/mol
New certification period: This material is valid until 01 January 2009.

SRM 2648a Propane in Nitrogen 5000 umol/mol
New certification period: This material is valid until 01 January 2009

REVISIONS continued…

SRM 2657a Oxygen in Nitrogen 2% umol/mol
New expiration date: This material is valid until 01 April 2008.

SRM 2659a Oxygen in Nitrogen 21% mol/mol
New expiration date: This material is valid until 01 April 2008.

SRM 3193 Aqueous Electrolytic Conductivity (Lot 012703)
New certification period: This material is valid until 27 November 2003

SRM 3195 Aqueous Electrolytic Conductivity (Lot 011804)
New certification period: This material is valid until 24 May 2004.

SRM 8412 Corn (Zea mays) Stalk
Corrected number of bottles given in the unit size description.

Expiration Dates Extended 5 Years for Rockwell Hardness SRMs:

SRM 2810 Rockwell C Scale Hardness Low Range with Serial Numbers 95N25001 to 95N25140: Dec. 8, 2007 (was Dec. 8, 2002)
SRM 2810 Rockwell C Scale Hardness Low Range with Serial Numbers 97C25501 to 97C25563: May 1, 2011 (was May 1, 2006)
SRM 2811 Rockwell C Scale Hardness Mid Range with Serial Numbers 95N45001 to 95N45140: Dec. 8, 2007 (was Dec. 8, 2002)
SRM 2811 Rockwell C Scale Hardness Mid Range with Serial Numbers 97C45501 to 97C45559: May 1, 2011 (was May 1, 2006)
SRM 2812 Rockwell C Scale Hardness High Range with Serial Numbers 95N63001 to 95N63140: Dec. 8, 2007 (was Dec. 8, 2002)
SRM 2812 Rockwell C Scale Hardness High Range with Serial Numbers 97C63513 to 97C63602: May 1, 2011 (was May 1, 2006)